TEST METHODS FOR DETERMINING THE SUITABILITY OF METAL ALLOYS FOR USE IN OXYGEN-ENRICHED ENVIRONMENTS

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ABSTRACT

Materials are more flammable in oxygen-enriched environments than in air. When the structural elements of a system containing oxygen ignite and burn, the results are often catastrophic, causing loss of equipment and perhaps even human lives. Therefore, selection of the proper metallic and nonmetallic materials for use in oxygen systems is extremely important. While test methods for the selection of nonmetallic materials have been available for many years, test methods for the selection of metal alloys have not been available until recently. Several test methods are presented that were developed recently at NASA's White Sands Test Facility (WSTF) to study the ignition and combustion of alloys, including the supersonic and subsonic speed particle impact tests, the frictional heating and coefficient-of-friction tests, and the promoted combustion test. These test methods are available for commercial use.

INTRODUCTION

Because nearly all metallic and non-metallic structural materials are flammable in oxygen-enriched atmospheres, costly fires have occurred in oxygen systems. For example, catastrophic oxygen-related fires have occurred in the space program, hindering mission success and costing human lives [1]. In the medical community, fires in operating rooms have occurred during surgery of the head and neck [2-5]. In industry, numerous fires have been reported in regulators and manual valves, causing severe system damage and human injury [6-8].

Furthermore, advancing technology is creating a demand for higher oxygen-use temperatures and pressures. NASA is investigating the use of liquid oxygen (LO₂) and liquid hydrogen (LH₂) as propellants for propulsion systems for a new generation of space-based orbital transfer vehicles (OTVs). The new design concept for the LH₂/LO₂ engines involves the use of gaseous oxygen (GO₂) at 533 K (500 °F) and 34.5 MPa (5000 psi) to drive the turbine in the LO₂ turbopump. Industry is considering the use of gas cylinders at up to 27.6 MPa (4000 psi). The use of oxygen at these higher temperatures and pressures causes a concern for engineers because it increases the risk of fire.

No absolute solution exists for controlling fire hazards in oxygen systems. However, the possibility of oxygen-related fires can be diminished with proper design practices and careful selection of materials. Guidance regarding proper design practices for oxygen systems and the selection of nonmetallic materials is available in the open literature [1,9-11]. Until recently, however, test methods and selection procedures for metals were not readily available.

The development of test methods for determining the suitability of metals in oxygen-enriched atmospheres began at NASA's White Sands Test Facility (WSTF) in the late 1970's. Several methods to determine the ignitability and combustion characteristics of metals have been developed. Ignition characteristics are determined using the particle impact, frictional heating, and coefficient-of-friction tests; and combustion characteristics are determined using the promoted combustion test [9,10]. This paper describes each of these test methods.

PARTICLE IMPACT TEST METHODS

Particle impact has been recognized as an ignition source in oxygen systems for several years [12-15]. Additionally, experience at WSTF has demonstrated that metal particles entrained in flowing oxygen can ignite valves, as seen in Figure 1. Two particle impact test methods, one operating at supersonic velocities and the other at subsonic velocities, have been developed at WSTF. The methods are used to determine the minimum temperature, pressure, and velocity at which particle impact ignition occurs.

Supersonic Particle Impact Test Method

The supersonic particle impact test chamber (Figure 2) has been described by Benz et al. [16] It comprises a gas inlet and flow straightener, a particle injector, a converging nozzle, a diverging nozzle, and a test sample mounted on a holder. GO_2 and the particle enter the inlet section and are accelerated to supersonic velocities as they pass through the converging and diverging nozzles. After flowing through a short constant-area section, the oxygen and the particle impact a sample made of the metal alloy being tested.

Typical supersonic impact of a 2000 μ m- (0.08-in)-diameter aluminum particle results are shown in Figure 3. As the test sample temperature is increased, the susceptibility to ignition by particle impact increases.

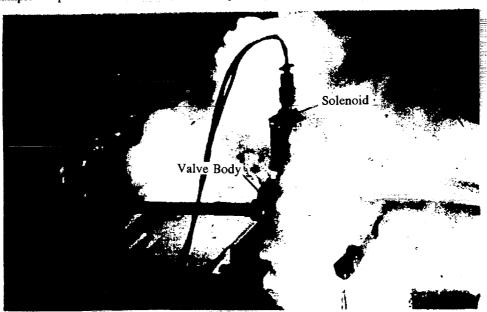


Figure 1
Valve Ignited by Metal Particles

Subsonic Particle Impact Test Method

The subsonic particle impact test chamber (Figure 4) has been described by Williams et al. [17]. It comprises a particle injector, a test sample, and a flow control orifice. Up to 5 g (0.01 lbs) of particles are injected into flowing oxygen and carried through the test chamber where they impact the test sample made from the metal alloy being tested. After impacting the sample, the oxygen and particles flow through holes on the sample periphery and are vented to the atmosphere through the flow control orifice.

Typical subsonic particle impact results are shown in Figure 5. For tests at ambient temperature with 2 g (0.004 lb) of iron powder, stainless-steel target ignitions do not occur until the particle velocity is increased to approximately 50 m/s (164 ft/s). When the temperature is increased to 360 to 450 K (188.6 to 350 °F), ignitions occur at 25 m/s (82 ft/s).

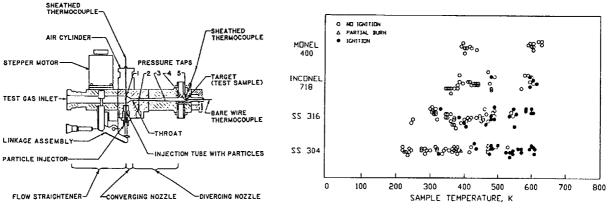
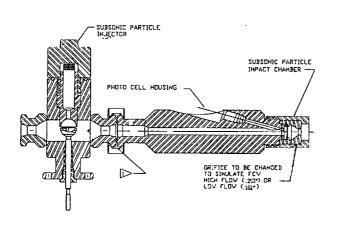


Figure 2
Supersonic Particle Impact Test Chamber

Figure 3
Typical Supersonic Particle Impact Results



100 s/w 75 Ignition of Stainless Steel Target Gas Velocity 50 25 No Ignition of Stainless Steel Target 0 300 350 400 450 500

Figure 4
Subsonic Particle Impact Test Chamber

Figure 5
Typical Subsonic Particle Impact Results

Temperature K

The supersonic and subsonic particle impact test methods are not patented, but are available for commercial use through NASA's technology transfer program.

FRICTIONAL HEATING TEST METHOD

When mechanical components of a system rub together, heat is generated by friction. This frictionally generated heat has been identified as the cause of many fires in oxygen-enriched environments. For example, the rotating machinery shown in Figure 6 ignited and burned when a rotating part rubbed against the housing as the result of a bearing failure. Additionally, bearings in the space shuttle main engine high-pressure oxygen turbopump have ignited because of frictional heating when they failed during off-limit tests. The frictional heating test method was developed to determine metal alloys' susceptibility to frictional ignition in oxygen.

The frictional heating test apparatus shown in Figure 7 has been described by Benz and Stoltzfus [18]. It comprises an electrical drive motor and transmission assembly, a high-pressure test chamber, and a pneumatic cylinder. A rotating shaft extends through the test chamber and is connected at one end to the drive assembly

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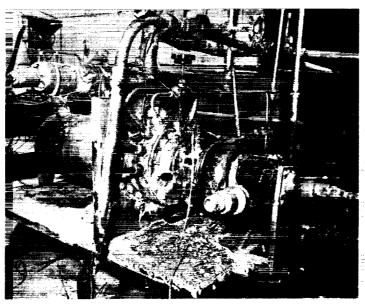
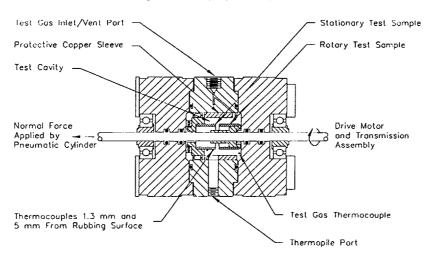


Figure 6
Rotating Machinery Ignited by Friction



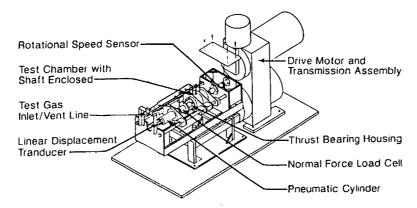


Figure 7
Frictional Heating Test Apparatus

and at the other end to the pneumatic cylinder. The rotating test specimen is mounted on the shaft, and the stationary test specimen is affixed to the test chamber. Tests up to 27.6 MPa (4000 psi) in GO₂ and 2 MPa (300 psi) in LO₂ can be conducted in this test apparatus.

Typical results of frictional heating tests are shown in Table 1. The results are presented in terms of the product of the loading pressure (P) and the rubbing velocity (v) required for ignition of the material pair.

Table 1
Results of Frictional Heating Tests in LO₂ and GO₂

MATERIAL		Pv Product		Reaction	
Stationary	Rotary	(W/m ²	x 10 ⁻⁸)		
		GOX	LOX	GOX	LOX
Inconel 718	Inconel 718	0.96-1.18	3.85	Yes	Yes
Monel K-500	Monel K-500	1.37-1.69	4.15-4.23	Yes	No
Inco MA754	Inco MA754	3.96-4.12	3.29-4.03	No	No
Haynes 214	Haynes 214	3.05	3.79-4.09	Yes	No
AMS 6278	AMS 6278	1.82	3.34	Yes	Yes
Al 6061T6	Al 6061T6	0.48	-	Yes	
Al 2219	Al 2219	-	1.72		Yes
Monel K-500	Kel-F	-	0.23-0.45		No
Monel K-500	Vespel SP21	-	1.52		Yes

"Sample deformation before ignition

The LO₂ and GO₂ frictional heating test methods are patented and are available for licensing by NASA.

COEFFICIENT-OF-FRICTION TEST METHOD

While frictional ignition data are available using the previously described test method, a need remains to understand the tribological behavior of metals in oxygen. A test apparatus is currently under development at WSTF for this purpose (see Figure 8).

The coefficient-of-friction test method comprises an electrical drive motor and transmission assembly, a pin-on-disk frictional contact device, and a high-pressure chamber. The drive motor and transmission assembly is similar to the one used in the frictional heating test. The pin-on-disk frictional contact device comprises a cylindrical test pin with a spherical end, a disk, two shafts that penetrate the chamber, and a load arm. The pin is mounted on the load arm, which is connected to the first shaft. A measured torque applied to this shaft by means of a pneumatic rotary actuator produces the normal contact force between the pin and the 5.1-cm-(2.0-in)-diameter test disk. The disk is mounted on the second shaft, which is connected to the drive motor assembly. As the pin contacts the rotating test disk, a frictional load is detected by a calibrated strain gauge mounted on the load arm. A 5.1-cm-(2.0-in)-diameter viewport in the chamber wall allows video coverage of the test. Tests at pressures up to 27.6 MPa (4000 psi) in GO₂ and 2 MPa (300 psi) in LO₂ can be conducted in this test system. Checkout tests are being conducted and a patent is pending on the coefficient-of-friction test method. It will be available for licensing once the patent is approved.

PROMOTED COMBUSTION TEST METHOD

To determine if an alloy can be safely used in an oxygen system, its flammability must be known. If it is not flammable in a configuration similar to that in which it is intended for use, it can be used safely. The promoted

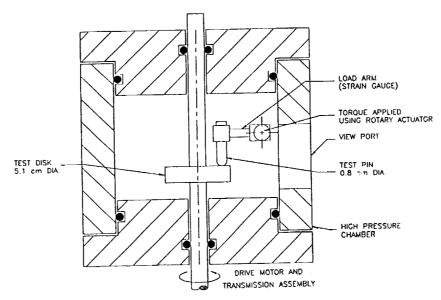


Figure 8
Coefficient-of-Friction Test Apparatus

combustion test measures flammability in terms of the minimum pressure required to support complete combustion of an alloy. This test method has been adopted by NASA as a standard for the selection of materials to be used in spacecraft. It is also being prepared as a standard by the American Society of Testing and Materials (ASTM) to determine the combustion behavior of metallic materials. The promoted combustion

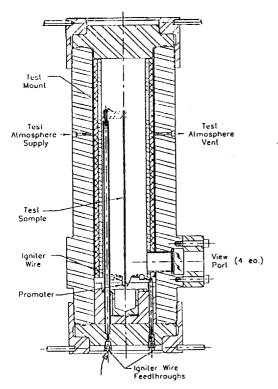


Figure 9
Promoted Combustion Test Apparatus

test apparatus shown in Figure 9 has been described by Stoltzfus et al. [19]. It comprises a cylindrical chamber, a copper liner and baseplate to protect the chamber from burning metal, and a sample mounting device. The chamber can be pressurized to 68.9 MPa (10,000 psia) and has four 5.1-cm- (2.0-in)-diameter viewports for sample observation and video recording. The test sample is held at the top in the sample mounting device and is ignited at the bottom by an aluminum or magnesium promotor. The promoter is ignited by an electrically heated wire.

Typical results of promoted combustion tests on several commonly used alloys are shown in Table 2. The table presents the minimum oxygen pressure required for complete combustion of 0.32-cm-(0.125-in)-diameter metal rods (threshold pressure). Nickel, copper, and their alloys are generally the least flammable. Iron-base alloys tend to be more flammable than the nickel- and copper-base alloys, but less flammable than the aluminum- and titanium-base alloys.

The promoted combustion test apparatus is patented and is available for licensing by NASA.

Table 2
Typical Promoted Combustion Test Results

Material	Threshold Pressure		
	MPa	(psia)	
Monel K-500	68.9	>10,000	
Inconel MA754	68.9	>10,000	
Haynes 214	68.9	>10,000	
Monel 400	68.9	>10,000	
Brass 360 CDA	68.9	> 10,000	
Nickel 200	55.2	> 8,000	
Inconel 600	20.7	3,000	
Inconel 625	20.7	3,000	
Inconel 718	6.9	1,000	
304 SS	6.9	1,000	
Aluminum 6061	0.68	100	
Aluminum 99.9%	0.17	25	
Ti-6Al-4V	0.007	1	

APPLICATION OF TEST METHODS

Johnson Space Center, Kennedy Space Center, Marshall Space Flight Center, and Lewis Research Center have used all these test methods to evaluate metals for use in oxygen systems. The results have been used to select metal alloys for the shuttle main propulsion system (MPS) oxygen flow control valves, the shuttle MPS high pressure oxygen turbopump bearings, advanced propulsion systems for the Space Exploration Initiative (SEI), and ground support equipment. The Langley Research Center has used particle impact data to select materials to use in the transpiration-cooled nozzle design for a wind tunnel.

The Department of Defense has used the test methods to determine the relative compatibility of aluminum alloys that may be used in cryogenic propellant tankage in future advanced launch systems. The data obtained have also been used to determine ignition and combustion hazards on aircraft main, backup, and emergency oxygen supply systems. Finally, the ground carts that are used to resupply aircraft oxygen systems on the flight line are being evaluated and new designs are being considered, based on the data from these test methods.

ASTM sponsored a test program that used the particle impact, frictional heating, and promoted combustion test methods to test alloys used in industrial oxygen systems. ASTM has published over 21 papers in their Standard Technical Publications containing data from these test methods [20-24]. ASTM has also prepared a training course entitled "Controlling Fire Hazards in Oxygen Systems" that makes extensive use of data from these methods. Additionally, several commercial companies have used these test methods to make material selections for applications ranging from aircraft turbine engines to compressor seal design.

These test methods are not only used by engineers for designing safe oxygen systems but they are also used for scientific research in the field of metals combustion. They have been used as tools for understanding the theories of ignition and combustion of metals and alloys [25-27]. It is anticipated that this understanding will aid in the development of new burn-resistant structural alloys for use in oxygen environments.

In summary, fire hazard in oxygen systems is a serious problem. However, a solution that minimizes this hazard exists using data from the test methods described in this paper. These methods have been used extensively in the past and are available for commercial use in the future.

REFERENCES

- 1) NFPA 53M. "Fire Hazards in Oxygen-Enriched Atmospheres." 1990 Edition in ASTM Standards
 Technology Training "Fire Hazards in Oxygen Systems." Edited by B. L. Werley, 1991.
- 2) de Richmond, A. L. "Laser Resistant Endotracheal Tubes--Protection Against Oxygen-Enriched Airway Fires During Surgery?" Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fifth Volume, ASTM STP 1111. Edited by J. M. Stoltzfus and K. McIlroy, American Society for Testing and Materials, Philadelphia, 1991.
- 3) Sidebotham, G. W., G. L. Wolf, J. Stern, and R. Aftel. "Endotracheal Tube Fires: A Flame Spread Phenomenon." Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fifth Volume, ASTM STP 1111. Edited by J. M. Stoltzfus and K. McIlroy, American Society for Testing and Materials, Philadelphia, 1991.
- 4) Bruley, M. E., and C. Lavanchy. "Oxygen-Enriched Fires During Surgery of the Head and Neck."

 Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth Volume, ASTM STP 1040. Edited by J. M. Stoltzfus, F. J. Benz, and J. S. Stradling, American Society for Testing and Materials, Philadelphia, 1989.
- 5) Simpson, J. I., G. L. Wolf, and G. A. Schiff. "The Oxidant O₂ (Helium) Index Flammability of Endotracheal Tubes." <u>Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth Volume, ASTM STP 1040</u>. Edited by J. M. Stoltzfus, F. J. Benz, and J. S. Stradling, American Society for Testing and Materials, Philadelphia, 1989.
- 6) Dicker, D. W. G. and R. K. Wharton. "A Review of Incidents Involving the Use of High-Pressure Oxygen from 1982 to 1985 in Great Britain" Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.
- 7) Barter, S. A. and L. W. Hillen. "Oxygen Fires, Materials Compatibility and System Contaminants"

 Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth

 Volume, ASTM STP 1040. Edited by J. M. Stoltzfus, F. J. Benz, and J. S. Stradling, American

 Society for Testing and Materials, Philadelphia, 1989.
- 8) Newton, B. E., R. K. Langford, and G. R. Meyer. "Promoted Ignition of Oxygen Regulators."

 Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth

 Volume, ASTM STP 1040. Edited by J. M. Stoltzfus, F. J. Benz, and J. S. Stradling, American

 Society for Testing and Materials, Philadelphia, 1989.
- 9) Bond, A.C., H. O. Pohl, N. H. Chaffee, W. W. Guy, C. S. Alton, R. L. Johnston, W. J. Castner, and J. S. Stradling. "Design Guide for High Pressure Oxygen Systems", NASA Reference Publication 1113.
- 10) Wegener, W., C. Binder, P. Hengstenberg, K. P. Herrmann, and D. Weinert. "Tests to Evaluate the Suitability of Materials for Oxygen Service." Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.
- 11) Lockhart, B. J., M. D. Hampton, and C. Bryan. "The Oxygen Sensitivity/Compatibility Ranking of Several Materials by Different Test Methods." Symposium on Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth Volume, ASTM STP 1040. Edited by J. Stoltzfus, F. J. Benz, and J. S. Stradling, American Society for Testing and Materials, Philadelphia, 1989.

- 12) Lapin, A. "Liquid and Gaseous Oxygen Safety Review." Vol. I-IV, NASA-CR-120922, APCI TM184, NASA Lewis Research Center, Cleveland, OH, June 1972.
- 13) Clark, A. F. and T. G. Hurst. AReview of the Compatibility of Structural Materials with Oxygen." AIAA Journal, Vol. 12, No. 4, April 1974, pages 441-454.
- 14) Wegener, W. "Investigations on the Safe Flow Velocity to be Admitted for Oxygen in Steel Pipe Lines." Stahl and Eisen, Vol. 84, No. 8, 1964, pages 469-475.
- 15) Williams, R. E. "The Combustion of Laser Ignited Zirconium." Final report NSG 518, NASA, Washington, DC, 1967.
- 16) Benz, F. J., R. E. Williams, and D. A. Armstrong. "Ignition of Metals and Alloys by High-Velocity Particles." <u>Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Second Volume. ASTM STP 910</u>. Edited by M. A. Benning, American Society for Testing and Materials, Philadelphia, 1986.
- 17) Williams, R. E., F. J. Benz, and K. McIlroy. "Ignition of Steel Alloys by Impact of Low-Velocity Iron/Iron Particles in Gaseous Oxygen." Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.
- 18) Benz, F. J., and J. M. Stoltzfus. "Ignition of Metals and Alloys in Gaseous Oxygen by Frictional Heating." Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Second Volume, ASTM STP 910. Edited by M. A. Benning, American Society for Testing and Materials, Philadelphia, 1986.
- 19) Stoltzfus, J. M., J. M. Homa, R. E. Williams, and F. J. Benz. "ASTM Committee G-4 Metals Flammability Test Program: Data and Discussion." Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.
- 20) Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: First Volume, ASTM STP 812, Edited by B. L. Werley, American Society for Testing and Materials, Philadelphia, 1983.
- 21) Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Second Volume, ASTM STP 910. Edited by M. A. Benning, American Society for Testing and Materials, Philadelphia, 1986.
- 22) Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.
- 23) Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fourth Volume, ASTM STP 1040. Edited by J. M. Stoltzfus, F. J. Benz, and J. S. Stradling, American Society for Testing and Materials, Philadelphia, 1989.
- 24) Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fifth Volume, ASTM STP 1111. Edited by J. M. Stoltzfus and K. McIlroy, American Society for Testing and Materials, Philadelphia, 1991.
- 25) Sato, J. and T. Hirano. "Fire Spread Limits Along Metal Pieces in Oxygen" Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Third Volume, ASTM STP 986. Edited by D. W. Schroll, American Society for Testing and Materials, Philadelphia, 1988.

- 26) Steinberg, T. A. and F. J. Benz. "Iron Combustion in Microgravity" Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fifth Volume, ASTM STP 1111. Edited by J. M. Stoltzfus and K. McIlroy, American Society for Testing and Materials, Philadelphia, 1991.
- 27) Sircar, S., H. Gabel, J. M. Stoltzfus, and F. J. Benz. "The Analysis of Metals Combustion Using a Real-Time Gravimetric Technique" Flammability and Sensitivity of Materials in Oxygen-Enriched Atmospheres: Fifth Volume, ASTM STP 1111. Edited by J. M. Stoltzfus and K. McIlroy, American Society for Testing and Materials, Philadelphia, 1991.